Notes

Structural Transformations of a Series of Tellurium−Iron Carbonylates and the Isolation of the Cubic Cluster [Te₄Fe₄(CO)₁₀(dppm)]

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Summary: The cluster growth from the small anionic cluster [TeFe₃(CO)₉]²⁻ to the double-cubic cluster [Te₁₀Fe₈(CO)₂₀]²⁻ was systematically established. Further treatment of [Te₁₀Fe₈(CO)₂₀]²⁻ with [Cu₂(dppm)₂(MeCN)₄]-[BF₄]₂ led to the formation of the new dppm-bridged cubic cluster [Te₄Fe₄(CO)₁₀(dppm)].

Introduction

Tellurium-rich metal compounds have attracted extensive attention, since tellurides exhibit unusual structural and reactivity patterns and are potentially precursors for new solid-state materials.[1,2] Recently, a series of novel tellurium-iron carbonylates have been synthesized and structurally characterized by several different methodologies. These interesting anionic clusters include [TeFe₃(CO)₁₂]²⁻,[3a] [TeFe₃(CO)₉]²⁻,[3,4] [Te₄Fe₅(CO)₁₄]²⁻,[5,6] [Te₆Fe₈(CO)₂₄]²⁻,[5] and [Te₁₀Fe₈(CO)₂₀]²⁻.[6] Nevertheless, their structural relationships and transformations have never been systematically studied. The reaction sequences in which the small molecules transform to larger clusters are of great importance to the extended inorganic solids.[7] As a consequence, develop-

ing and understanding the conversion of small clusters to larger ones is of great interest. In this paper, we demonstrate a facile stepwise cluster growth from [TeFe₃(CO)₉]²⁻ to [Te₁₀Fe₈(CO)₂₀]²⁻. The reactions of [Te₁₀Fe₈(CO)₂₀]²⁻ with electrophiles are described as well, from which the cubic cluster [Te₄Fe₄(CO)₁₀(dppm)] is obtained and compared with the double-cubic cluster [Te₁₀Fe₈(CO)₂₀]²⁻.

Experimental Section

All reactions were performed under an atmosphere of pure nitrogen by using standard Schlenk line techniques.[8] Solvents were purified, dried, and distilled under nitrogen prior to use. K₂TeO₃·H₂O (Alfa), TeO₂ (Strem), Fe(CO)₅ (Aldrich), and bis-(diphenylphosphino)methane (dppm; Acros) were used as received. Infrared spectra were recorded on a Jasco 700 IR or a Perkin-Elmer Paragon 500 spectrometer using CaF₂ liquid cells. Mass spectra were obtained on a Finnigan TSQ700 mass spectrometer. Elemental analyses were performed on a Perkin-Elmer 2400 analyzer at the NSC Regional Instrumental Center at National Taiwan University. ¹H NMR spectra were taken on a Varian 200 (200 MHz) instrument. [TeFe₃(CO)₉]²⁻,[3,4] Te₂Fe₃(CO)₉,[9] and [Cu₂(dppm)₂(MeCN)₄][BF₄]₂[10] were prepared by the published methods.

Formation of [Et₄N]₂[Te₄Fe₅(CO)₁₄]. To a mixture of 0.239 g (0.296 mmol) of [Et₄N]₂[TeFe₃(CO)₉] and 0.100 g (0.148 mmol) of Te₂Fe₃(CO)₉ was added 20 mL of THF. After being stirred for 3 days at room temperature, the solution was filtered and solvent was removed under vacuum. The residue was washed with hexanes and ether and then extracted with CH₂Cl₂ to give 0.110 g (0.076 mmol) of [Et₄N]₂[Te₄Fe₅(CO)₁₄] (26% based on [Et₄N]₂[TeFe₃(CO)₉]). IR (ν_CO, MeCN) for [Et₄N]₂[Te₄Fe₅(CO)₁₄]: 2025 w, 2008 s, 1975 vs, 1940 m cm⁻¹.

Formation of [Et₄N]₂[Te₆Fe₈(CO)₂₄]. To a mixture of 0.100 g (0.069 mmol) of [Et₄N]₂[Te₄Fe₅(CO)₁₄] and 0.086 g (0.127 mmol) of Te₂Fe₃(CO)₉ was added 40 mL of CH₂Cl₂. The solution turned reddish brown immediately and was filtered, and solvent was removed under vacuum. The residue was washed with hexanes and ether and then extracted with CH₂Cl₂ to give 0.110 g (0.051 mmol) of [Et₄N]₂[Te₆Fe₈(CO)₂₄]

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(74% based on [Et$_4$N]$_2$[Te$_4$Fe$_5$(CO)$_{14}$]). IR (ν_{CO}, CH$_2$Cl$_2$) for [Et$_4$N]$_2$[Te$_6$Fe$_8$(CO)$_{24}$]: 2026 s, 2004 vs, 1956 m (br) cm^{-1}.

Formation of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$]. To a mixture of 3.20 g (1.46 mmol) of [TMBA]$_2$[Te$_6$Fe$_8$(CO)$_{24}$] and 0.967 g (7.58 mmol) of Te powder was added 40 mL of THF. After being stirred and heated at 50 °C for 3 days, the solution was filtered and solvent was removed under vacuum. The residue was washed with hexanes and ether and then extracted with CH$_2$Cl$_2$ to give 2.24 g (0.867 mmol) of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$] (60% based on [TMBA]$_2$[Te$_6$Fe$_8$(CO)$_{24}$]). IR (ν_{CO}, CH$_2$Cl$_2$): 2026 s, 2015 sh, 1979 s, 1924 w cm^{-1}. Anal. Calcd (found) for [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$]: C, 18.57 (18.59); H, 1.24 (1.44); N, 1.08 (0.95).

Reaction of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$] with Na/Naphthalene. To a sample of 0.297 g (0.115 mmol) of [TMBA]$_2$[Te$_{10}$-Fe$_8$(CO)$_{20}$] in 15 mL of THF was added 1.2 mL of a solution which was prepared from 0.349 g (0.015 mol) of Na and 1.94 g (0.015 mol) of naphthalene in 40 mL of THF. The mixed solution was stirred for 10 h and filtered, and solvent was removed under vacuum. The residue was washed with hexanes and then extracted with CH$_2$Cl$_2$ to give 0.11 g (0.050 mmol) of [TMBA]$_2$[Te$_6$Fe$_8$(CO)$_{24}$] (43% based on [TMBA]$_2$[Te$_{10}$-Fe$_8$(CO)$_{20}$]). IR (ν_{CO}, CH$_2$Cl$_2$) for [TMBA]$_2$[Te$_6$Fe$_8$(CO)$_{24}$]: 2027 m, 2006 vs, 1959 m, br cm^{-1}.

Reaction of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$] with MeSO$_3$CF$_3$. To a sample of 0.203 g (0.079 mmol) of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$] in 10 mL of THF was added 0.04 mL (0.35 mmol) of MeSO$_3$-CF$_3$. The mixed solution was stirred for 3 h and filtered, and solvent was removed under vacuum. The residue was extracted with hexanes to give the known butterfly complex 0.02 g (0.034 mmol) (Fe$_2$(CO)$_6$(μ-TeMe)$_2$,[11] which was identified by IR and mass spectroscopy.

Formation of [Te$_4$Fe$_4$(CO)$_{10}$(dppm)]. To a mixture of 0.774 g (0.300 mmol) of [TMBA]$_2$[Te$_{10}$Fe$_8$(CO)$_{20}$] and 0.369 g (0.300 mmol) of [Cu$_2$(dppm)$_2$(MeCN)$_4$][BF$_4$]$_2$ was added 30 mL of THF. After being stirred and heated at 50 °C for 40 h, the solution was filtered and solvent was removed under vacuum. The residue was washed with MeOH and ether and then extracted with THF to give 0.25 g (0.169 mmol) of [Te$_4$-Fe$_4$(CO)$_{10}$(dppm)] (28%). IR (ν_{CO}, CH$_2$Cl$_2$) for [Te$_4$Fe$_4$(CO)$_{10}$-(dppm)]: 2053 s, 2032 vs, 1994 vs, 1940 w cm^{-1}. Anal. Calcd (found) for [Te$_4$Fe$_4$(CO)$_{10}$(dppm)]: C, 29.19 (29.10); H, 1.63 (1.81). ^{1}H NMR (CDCl$_3$, 298 K): δ 7.55−7.22 (m, 20H), δ 4.04 (t, 2H, J_{P-H} = 10 Hz). Mp: 319-320 °C dec. This complex is soluble in ether, CH$_2$Cl$_2$, and THF.

X-ray Structural Characterization of [Te$_4$Fe$_4$(CO)$_{10}$-(dppm)]·CH$_2$Cl$_2$ (1). A summary of selected crystallographic data for [Te$_4$Fe$_4$(CO)$_{10}$(dppm)]·CH$_2$Cl$_2$ (1) is given in Table 1. Data collection was carried out on a Nonius CAD-4 diffractometer using graphite-monochromated Mo Kα radiation at 25 °C employing the $\theta/2\theta$ scan mode. A ψ-scan absorption correction was made.[12] The crystal was mounted on glass fibers with epoxy cement. Data reduction and structural refinement were performed using the NRCC-SDP-VAX packages,[13] and atomic scattering factors were taken from ref 14.

Reddish black crystals of **1** suitable for X-ray analysis were grown from hexane/CH$_2$Cl$_2$ solutions. A total of 7842 unique reflections were collected and corrected for absorption and decay. The structure was solved by the heavy-atom method and refined by least-squares cycles. All non-hydrogen atoms were refined with anisotropic thermal parameters. Full-matrix least-squares refinement of **1** led to convergence with R = 3.4% and R_w = 3.2% for those reflections with I > 2.0 $\sigma(I)$.

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Table 1. Selected Crystallographic Data^a for [Te$_4$Fe$_4$(CO)$_{10}$(dppm)]·CH$_2$Cl$_2$ (1)

empirical formula	C$_{36}$H$_{24}$Cl$_2$Fe$_4$O$_{10}$P$_2$Te$_4$
fw	1483.21
cryst syst	triclinic
space group	P$\bar{1}$
a, Å	11.724(5)
b, Å	13.836(5)
c, Å	15.116(5)
α, deg	84.97(3)
β, deg	86.70(3)
γ, deg	66.24(3)
V, Å^3	2235(2)
D(calcd), Mg m^{-3}	2.204
abs coeff, cm^{-1}	40.837
T_{min}/T_{max}	0.90/1.00
residuals: R; R_w^{b}	0.034; 0.032

^{a} All data were obtained from a Nonius (CAD-4) diffractometer with Mo Kα radiation (λ = 0.7107 Å) at 25 °C. ^{b} The functions minimized during least-squares cycles were $R = \sum|F_o - F_c|/\sum F_o$ and $R_w = [\sum w(F_o - F_c)^2/\sum w(F_o)^2]^{1/2}$.

Table 2. Selected Bond Distances (Å) and Bond Angles (deg) for [Te$_4$Fe$_4$(CO)$_{10}$(dppm)]·CH$_2$Cl$_2$ (1)

(A) Distances			
Te(1)−Fe(1)	2.615(2)	Te(1)−Fe(2)	2.607(2)
Te(1)−Fe(3)	2.619(2)	Te(2)−Fe(1)	2.620(2)
Te(2)−Fe(2)	2.623(2)	Te(2)−Fe(4)	2.608(2)
Te(3)−Fe(1)	2.605(2)	Te(3)−Fe(3)	2.619(2)
Te(4)−Fe(2)	2.621(2)	Te(4)−Fe(3)	2.621(2)
Te(4)−Fe(4)	2.613(2)	Te(3)−Fe(4)	2.625(2)

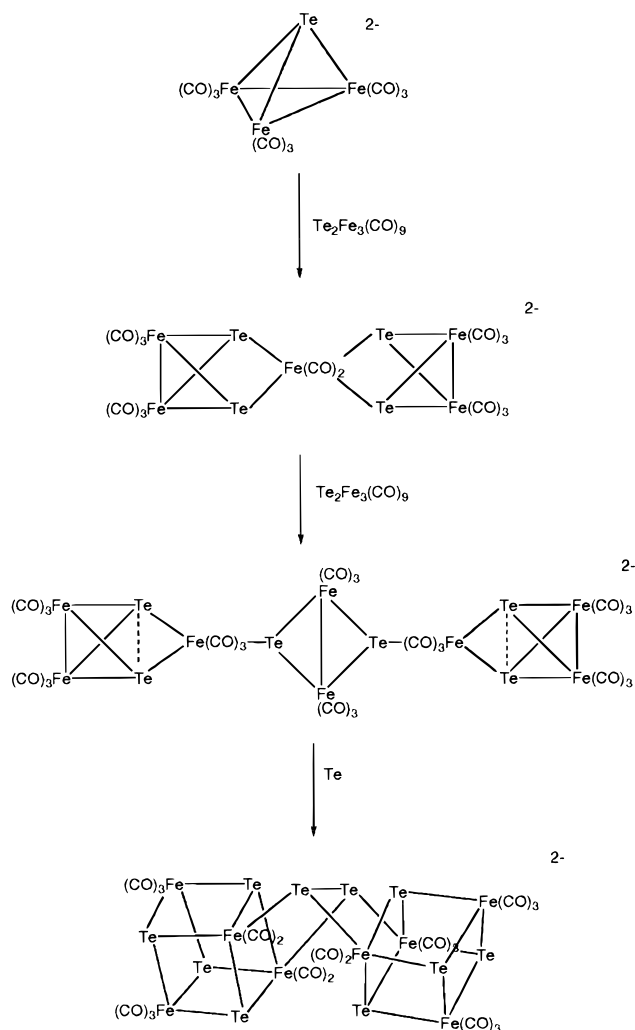| (B) Bond Angles			
Fe(1)−Te(1)−Fe(2)	92.95(6)	Fe(1)−Te(1)−Fe(3)	97.88(6)
Fe(2)−Te(1)−Fe(3)	97.40(5)	Fe(1)−Te(2)−Fe(2)	92.47(6)
Fe(1)−Te(2)−Fe(4)	96.66(5)	Fe(2)−Te(2)−Fe(4)	98.64(6)
Fe(1)−Te(3)−Fe(3)	98.14(6)	Fe(1)−Te(3)−Fe(4)	96.60(5)
Fe(3)−Te(3)−Fe(4)	95.51(6)	Fe(2)−Te(4)−Fe(3)	96.99(5)
Fe(2)−Te(4)−Fe(4)	98.55(6)	Fe(3)−Te(4)−Fe(4)	95.71(6)
Te(1)−Fe(1)−Te(2)	86.23(5)	Te(1)−Fe(1)−Te(3)	82.04(5)

Selected bond distances and angles of **1** are listed in Table 2. Additional crystallographic data are available as Supporting Information.

Results and Discussion

The previous study[3,4] showed that the reaction of K$_2$TeO$_3$ with 3 equiv of Fe(CO)$_5$/KOH in MeOH led to the formation of the open cluster [TeFe$_3$(CO)$_{12}$]$^{2-}$, which then decarbonylated to generate the tetrahedral complex [TeFe$_3$(CO)$_9$]$^{2-}$. However, when K$_2$TeO$_3$ reacted with 1 equiv of Fe(CO)$_5$/KOH in MeOH, the large cluster anion [Te$_6$Fe$_8$(CO)$_{24}$]$^{2-}$ was formed.[4] These results indicated that the Te−Fe−CO system is very versatile, and different stoichiometries and subtle changes of the reaction conditions could give rise to different outcomes. These prompted us to explore the cluster transformations and to attempt to elucidate the cluster growth processes in this system.

Cluster Growth Processes. The cluster growth processes are summarized in Scheme 1. When the tetrahedral cluster [TeFe$_3$(CO)$_9$]$^{2-}$ was treated with Te$_2$-Fe$_3$(CO)$_9$, the mediun-sized cluster [Te$_4$Fe$_5$(CO)$_{14}$]$^{2-}$ was obtained. One can envision [Te$_4$Fe$_5$(CO)$_{14}$]$^{2-}$ to be composed of the anion [Te$_2$Fe$_2$(CO)$_6$]$^{2-}$ and one Te$_2$Fe$_3$-(CO)$_9$ with the loss of one CO, which seems to have no direct relevance to [TeFe$_3$(CO)$_9$]$^{2-}$ and Te$_2$Fe$_3$(CO)$_9$. Therefore, the formation of [Te$_4$Fe$_5$(CO)$_{14}$]$^{2-}$ can be regarded as resulting from complicated bond breakage and rearrangement processes. It was more interesting
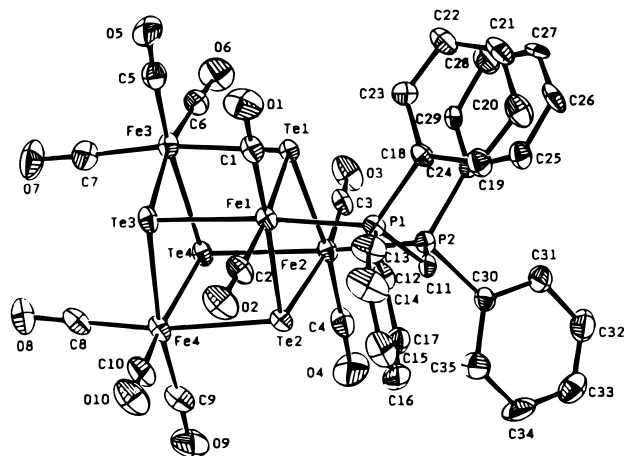
Scheme 1

Figure 1. ORTEP diagram showing the structure and atom labeling for **1**.

indicate that the cluster formation is gradual and may be predictable and also provide some useful information on further cluster expansion reactions.

Reactivity of $[Te_{10}Fe_8(CO)_{20}]^{2-}$. Since the double-cubic cluster $[Te_{10}Fe_8(CO)_{20}]^{2-}$ is structurally interesting and can be obtained easily from $[Te_6Fe_8(CO)_{24}]^{2-}$, further reactivity studies of $[Te_{10}Fe_8(CO)_{20}]^{2-}$ were therefore carried out. It was found that $[Te_{10}Fe_8(CO)_{20}]^{2-}$ can be reconverted to $[Te_6Fe_8(CO)_{24}]^{2-}$ upon reduction with Na/naphthalene (eq 1). To test the basic sites of

$$[Te_6Fe_8(CO)_{24}]^{2-} \underset{\text{Na/naphthalene}}{\overset{4Te}{\rightleftarrows}} [Te_{10}Fe_8(CO)_{20}]^{2-} \quad (1)$$

$[Te_6Fe_8(CO)_{24}]^{2-}$, we treated $[Te_{10}Fe_8(CO)_{20}]^{2-}$ with the methylation agent $MeSO_3CF_3$. However, the reaction proceeded with severe bond breakage and formation to give the known butterfly complex[11] $Fe_2(CO)_6(\mu\text{-TeMe})_2$. When $[Te_{10}Fe_8(CO)_{20}]^{2-}$ was treated with $[Cu_2(dppm)_2(MeCN)_4][BF_4]_2$, the dppm-bridged cubic cluster $[Te_4Fe_4(CO)_{10}(dppm)]$ (**1**) was obtained (eq 2). Cluster **1**

$$[Te_{10}Fe_8(CO)_{20}]^{2-} + [Cu_2(dppm)_2(MeCN)_4][BF_4]_2 \rightarrow$$
$$[Te_4Fe_4(CO)_{10}(dppm)] \quad (2)$$
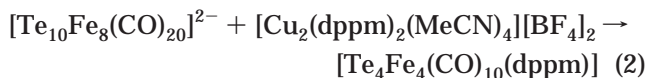
can be considered to result from the oxidative fragmentation of $[Te_{10}Fe_8(CO)_{20}]^{2-}$ by the Cu(I) complex followed by the chelation of the dppm ligand. Direct reaction of $[Te_{10}Fe_8(CO)_{20}]^{2-}$ with dppm failed to produce cluster **1**.

The reactivity investigation showed that the double-cubic cluster tended to undergo fragmentation to give the cubic or even smaller fragments depending upon the incoming reagents. However, the potential of $[Te_{10}Fe_8(CO)_{20}]^{2-}$ to serve as a cubic precursor is promising, and further study of its cluster expansion is now in progress.

Structure of $[Te_4Fe_4(CO)_{10}(dppm)]\cdot CH_2Cl_2$ (1). $[Te_4Fe_4(CO)_{10}(dppm)]$ was structurally characterized by single-crystal X-ray diffraction. As shown in Figure 1,

to note that when $[Te_4Fe_5(CO)_{14}]^{2-}$ further reacted with $Te_2Fe_3(CO)_9$, the large cluster $[Te_6Fe_8(CO)_{24}]^{2-}$ was cleanly yielded. This cluster growth process is quite reasonable, because $[Te_6Fe_8(CO)_{24}]^{2-}$ can be approximately viewed as one $[Te_4Fe_5(CO)_{14}]^{2-}$ anion and one $Te_2Fe_3(CO)_9$.

Utilizing this methodology, we wondered whether the larger cluster can be obtained when $[Te_6Fe_8(CO)_{24}]^{2-}$ was treated with $Te_2Fe_3(CO)_9$ or Te or other Fe sources. This is indeed the case. When $[Te_6Fe_8(CO)_{24}]^{2-}$ was treated with 4 equiv of Te, the double-cubic cluster $[Te_{10}Fe_8(CO)_{20}]^{2-}$ was obtained in good yield. The double-cubic cluster was previously reported to be generated from the reaction of $Fe(CO)_5$ with "Zintl" ion Te_4^{2-} and excess Te. The route we describe here from $[Te_6Fe_8(CO)_{24}]^{2-}$ to $[Te_{10}Fe_8(CO)_{20}]^{2-}$ is convenient and high yielding. From a structural viewpoint, there is no obvious relationship between these two clusters; however, the structural transformation is possible by adjustment of the appropriate stoichiometry.

In this study, the facile processes of the cluster growth from the small $[TeFe_3(CO)_9]^{2-}$ to the large $[Te_{10}Fe_8(CO)_{20}]^{2-}$ are established. Other types of cluster expansion processes have been seen in our previous studies of Se−Fe and E−Mn (E = S, Se) systems.[15,16] In this case, larger cluster skeletons are observed probably due to the greater size of the Te atom. The results all

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the Te_4Fe_4 core exhibits a cubic geometry with the dppm ligand bridging across two Fe atoms. Each iron atom in **1** is octahedrally coordinated and obeys the 18-electron count. The Te−Fe distance averages 2.616 Å and is close to those of the structurally related double-cubic cluster $[Te_{10}Fe_8(CO)_{20}]^{2-}$ (2.62 Å) and the cubic clusters $[Te_4Fe_4(SPh)_4]^{3-}$ (2.63 Å),[17] $[Te_4Fe_4(TePh)_4]^{3-}$ (2.622 Å),[18] and $[Te_4Fe_4(S^iPr)_4]^{3-}$ (2.627 Å).[19] In **1**, the internal angles in the Te_4Fe_4 cube are between 82.04 and 98.64°, close to 90°, indicative of a slight distortion from the ideal cube. This is different from the previously characterized cubes $[Te_4Fe_4(EPh)_4]^{3-}$ (E = S, Te),[17,18] which contain angles deviating greatly from 90°, probably due to the bridging effect of the dppm ligand on the cubic structure.

Supporting Information Available: Complete listings of crystallographic data, atomic positional parameters, bond distances and angles, and anisotropic thermal parameters of cluster **1** (7 pages). Ordering information is given on any current masthead page.

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